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Technology Transfer of the float polishing process from Japan has begun. Initially crystalline quartz was float polished. All of this work was reported at the Science of Optical Finishing Conference at Monterey, California. Photo-acoustic spectroscopy measurements indicated that float polishing removed a substantial portion of subsurface damage. Next, Corning 7940 substrates were float polished. After 230 μm of material had been removed through float polishing, there were no signs of any surface defects. Future directions to further develop float polishing for optical surfaces are discussed.

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Report AFOSR-90-0145

PRECISION FLOAT POLISHING

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Introduction

In 1989 a float polishing machine was purchased by UNM from Toyoda Machine Tools under AFOSR-89-0337 contract. The machine was purchased (1) to perform a technology transfer of the float polishing technique invented in Japan by Dr. Y. Namba to finish electronic components without damage and contamination, and (2) to investigate the fundamental parameters influencing float polishing in order to achieve low scatter, smooth, damage-free surfaces.

The machine was delivered in November 1989 and was installed the following month. This report will include the technical effort between 1 February 1990 and 31 January 1991 under contract AFOSR-90-0143.

Learning Period:

The first three months of this period were devoted to learning how to cut the tin and copper laps, how to fabricate the proper grooves, and how to deburr the lap prior to polishing. Because the optical part being polished only floats approximately one micron above the lap, it is essential that all metal burrs larger than 0.5 micron be removed. The procedure to machine and deburr the lap properly was developed during this period. Polishing then proceeded.

Polishing of Crystalline Quartz:

Crystalline quartz was chosen for polishing based upon previous studies using a rather crude, single spindle polishing machine. The float polishing technique was utilized in the fabrication of disc-shaped quartz resonators, having a diameter of 6.35 mm and a polished thickness of 104μm, to increase the fracture strength of the resonators. We were able to remove all the subsurface damage that manifests itself in lower stress failures. The stress fracture threshold was increased up to 4-10 times that of conventionally polished samples (approaching the bulk value). All of this work was reported at the Science of Optical Finishing Conference at Monterey, California on June 10-12, 1990. A copy of that paper is included in Appendix A.

In an attempt to fully quantify the surface roughness and the presence of sub-surface damage resulting from the polishing process, natural crystalline quartz substrates were polished and analyzed.

Natural quartz substrates, 25.4 mm in diameter, were polished to a thickness of 0.9 mm. The substrates were fixed with wax in a hexagonal pattern on a base that weighed 2000 gm. The substrates were first float polished on a diamond turned Cu lap with a 2% (by weight) TiO_2-deionized H_2O slurry. The TiO_2 particle size is approximately 1000 Å in diameter. The removal rate in this procedure was close to 1μm/hr., as measured by a digital micrometer. Approximately 10μm were removed from one face (Side A) and 100μm from the opposite face (Side B) of each sample. The substrates were then float polished with a slurry of the same composition as above on a Sn lap for 4 hr, and finally polished on a Sn lap with a 2% (by weight) 70 Å fumed SiO_2-deionized H_2O slurry. The slurry temperature was controlled at 20°C to prevent any lap warpage from occurring due to temperature changes during the polishing process. The rotational rate of the substrates and the lap were set at 75 rpm. The removal rate in this procedure was extremely slow and not measurable by standard techniques. The wax residue was removed by soaking in 111 tri-chloroethane, and an enamel paint mark was placed on the edge to distinguish the two sides. The substrates were inspected for overall surface quality using a Nomarski microscopy. On Side A there were still many imperfections in the surface. Side B had fewer imperfections, but not all imperfections had been removed.

Photoacoustic spectroscopy using surface acoustic wave (SAW) generation was performed on both surfaces of the samples to determine the surface absorption. This absorption is determined by the properties of approximately the first 50μm of material; the surface absorption is indicative of the presence of substoichiametric material or incorporated impurities in this layer. Photoacoustic spectroscopy is a well-known technique used for investigating physical properties of various media.[1] A system for

SAW detection is described in reference.[2] A pulsed optical beam in a one-dimensional grid pattern is imaged on the sample. The nonradiative fraction of the absorbed radiation results in a local rise in temperature and a corresponding decrease in the local density. This local thermal gradient propagates as a surface wave and is detected by a piezoelectric transducer coupled to the sample. The amplitude of this signal is proportional to the absorbed nonradiative fraction of power. SAW measurements of the two sides of a float-polished quartz sample are shown in Table 1.

Table 1

Surface	Photo-acoustic signal
Side A	1.3 ppm
Side B	0.4 ppm

The photoacoustic signal of side B is approximately one third that of side A. In fact the 0.4 ppm is very near the noise floor of the instrument. Our experience with photoacoustic absorption of conventionally polished optical components is that absorption levels are usually 1ppm or larger. Note that the photoacoustic signal did not vary on side B when the laser beam was incident on different regions of the surface. The absorption was the same when examining a region of a perfect surface area and when there were surface imperfections as observed by Nomarski microscopy. Because of this, it is believed that the surface imperfections that were observed are due to crystalline imperfections and not due to the sawing and lapping operations. In an effort to obtain better quality substrates, polishing of Corning 7940 fused silica was begun.

Corning 7940 Polishing:

Polished samples of Corning 7940 fused silica, fabricated by General Optic, were selected for float polishing. Initially the 1-inch samples were closely examined under

the Nomarski microscope. The surfaces appeared to be very good with no observable surface imperfections. These substrates were then float polished with a 2% (by weight) TiO_2-deionized H_2O slurry. The particle size of the TiO_2 is approximately 1000 Å in diameter. The rotational rate of the substrates and the lap were 80 rpm with a removal rate of approximately 1μm per hour. After 125μm were removed from the surface, defects were visible on the surface in Nomarski microscopy as shown in Fig. 1. There are randomly distributed defects on the surface. Also there seem to be defects systematically distributed in an arc having a radius of curvature of 2 inches ($\simeq$ 40 mils actual size) in the enlarged picture. Two microns of material were removed by further polishing. Most of the defects that were visible in Fig. 1 have now been removed, and new defects appear as indicated in Fig. 2. Notice the circular arrangement of defects approximately 0.25 inches in radius ($\simeq$ 5 mils actuai size). Another cluster of defects in a circular pattern (approximately 0.9 inches in radius) ($\simeq$ 18 mils actual size) is shown in Fig. 3. Figures 4 through 22 show a progression of defects that were observed in Fig. 3 as additional material is removed by float polishing. It appears that as material is removed, damage is removed in some areas but in other areas the damage increased, presumably the result of material fracture. This fracture may be due to stress in the material or to the shape of the microcracks in the material. When 230μm of material had been removed from the surface, there were no signs of any surface defects.

The samples were then float polished for 12 hours with 70Å fumed silica to obtain a smoother surface. Surface roughness measurements were taken of the three samples. These are shown in Table 2.

Table 2

Wyco Optical Profilometer

RMS Surface Roughness Measurements (Å)

Limit of Resolution 3.5 Å

218 Hour 1000 Å Titanium Oxide Polish Followed by

12 Hour 70 Å Fumed Silica Polish

Sample	measurement 1	measurement 2	measurement 3	average
Control	2.80	3.04	2.41	2.75
C20	4.99	6.11	3.27	4.79
C90	5.54	4.37	3.17	4.36
C109	6.43	3.33	3.94	4.57

Future Directions

Producing state-of-the-art optical quality surfaces of fused silica is the present goal of this project. To obtain an optical quality, the figure, surface imperfections, and subsurface damage of the optic are critical issues to be addressed. We plan to grind and float polish samples of rough-cut Corning 7940 fused silica with 1- and 2-inch diameters. Investigating the fundamental parameters of grinding and float polishing and how they influence the figure, surface, and subsurface damage on the samples will begin.

In particular, we will investigate the ζ-potential of a surface and the effects of altering the potential during grinding and polishing. There is a correlation between the ζ-potential of a substrate/slurry combination and the wear rate of the substrate. The wear rate is strongly correlated with the manner in which the surface is ground and polished, and in particular, the amount of subsurface damage that is produced. The ζ-potential is determined by several things including the substrate solubility, and the

electrolytic nature, pH level, and dielectric constant of the slurry. In our initial steps, we will vary the ζ-potential during grinding and assess the impact on the surface quality which is produced.

We will also investigate a more convenient method to assess the subsurface damage of a substrate. We will incorporate a technique developed by Paul Temple while at the Naval Weapons Center, which is called total internal reflectance microscopy (TIRM). We have taken initial steps toward this that show great promise for the technique to be a semi-quantitative, convenient diagnostic. This will provide rapid feedback for us to investigate polishing.

References

1. A. C. Tam, "Applications of Photoacoustic Sensing Techniques," Rev. Mod. Phys., 58(2), 381 (1986).

2. M. Y. A. Raja, D. W. Reicher, S. R. J. Brueck, J. R. McNeil, D. E. Oates, "High Sensitivity Surface-Photoacoustic Spectroscopy," Optics Letters, 15(1), 66 (1990).

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 1 (125 μm Removal)

Figure 2 (127 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 3 (152 μm Removal)

Figure 4 (156 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 5 (162 μm Removal)

Figure 6 (168 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 7 (170 μm Removal)

Figure 8 (176 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 9 (188 μm Removal)

Figure 10 (190 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 11 (196 μm Removal)

Figure 12 (198 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 13 (202 μm Removal)

Figure 14 (208 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 15 (214 μm Removal)

Figure 16 (218 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH
50 X

Figure 17 (220 μm Removal)

Figure 18 (224 μm Removal)

GENERAL OPTIC 7940
1000 Å TiO_2 FLOAT POLISH

Figure 19 (226 μm Removal 50 X)

Figure 20 (226 μm Removal 100 X)

GENERAL OPTIC 7940
100 X

Figure 21 (230 μm Removal)
1000 Å TiO_2 Float Polish

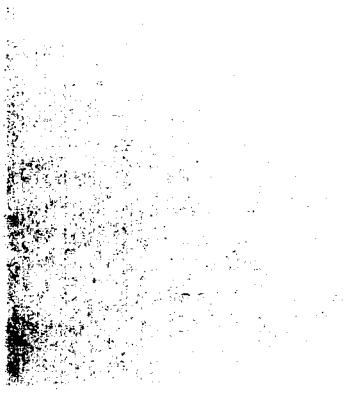

Figure 22 (< 1 μm Removal)
70 Å Fumed Silica Float Polish

APPENDIX A

FLOAT POLISHED QUARTZ SUBSTRATES *

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Float Polishing is a unique polishing technique developed in Japan, that produces supersmooth damage-free surfaces for electronic, optical and magnetic applications. Crystalline materials such as quartz, silicon and sapphire, polycrystalline materials such as ferrite and zinc sulfide, and amorphous materials such as fused silica and zerodur have been polished using this technique. Float polished substrates have a typical surface roughness of approximately 2 Å, with a flatness of $\lambda/20$. Float polishing is used commercially in the fabrication of ferrite recording heads, sapphire crowns for watches and quartz resonators.

In float polishing a loaded substrate and a precision machined lap are submerged in a slurry composed of deionized water and a measure of polishing powder. The experimental apparatus is shown in Figure 1. The sample and the lap are rotated in the

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same circular direction, and under equilibrium a fluid layer isolates the substrate from the lap. The thickness of this fluid layer is on the order of 1 μm. The dimension of polishing particles in the slurry is far less than this fluid layer thickness, and polishing action results from the impingement of the particles on the substrates. If the dimension of the polishing particles in the slurry is equal or greater than the fluid layer thickness, then mild abrasive action results and the removal rate exceeds that in float polishing. The variables of the removal rate are the load on the substrate, the type of slurry used, the lap material, the rotation rate, and the pH and temperature of the slurry. The flatness of the lap is essentially transferred to the substrate, and thus is also an important parameter. In the polishing operation, the slurry temperature increases and could cause lap warpage, affecting flatness. To prevent this the slurry temperature is usually controlled by a temperature controlled recirculator. There is normal lap wear that occurs if the polishing particles are large, and the lap is recut periodically.

Float polishing research at the University of New Mexico is conducted on the SP-46 polishing machine manufactured by Toyoda Corporation of Japan. This machine has several unique features. The lap size is large enough to polish approximately 6 in substrates. The lap itself is mounted on a hydraulically-stabilized spindle with a very high rotational accuracy. The lap material is typically tin, nylon or copper, and the lap is cut on the machine using natural diamond tools for a burr-free, precision finish. The spindle accuracy is also transferred to the lap. The maximum rotation speeds of the substrate and the lap are 200 rpm, and the rotation and the tooling operation in lap machining are microprocessor controlled. The polishing environment is maintained at class 100 cleanness because any particle larger than a fraction of a micron can cause surface scratching. The slurry temperature is controlled by circulating the slurry through a copper heat exchanger coil immersed in a constant temperature bath.

In previous research the float polishing technique was utilized in the fabrication of disc shaped quartz resonators, having a diameter of 6.35 mm and a polished thickness

of 104 μm to increase the fracture strength of the resonators. Some of the resonators had a stress-failure threshold approaching the bulk value of quartz. The resonators were purchased as AT-cut crystalline quartz blanks with the x-orientation marked by a flat and the easy-cleavage plane aligned at an angle 55 ° from the z-orientation. The substrates had a 3.0 μm CeO_2 finish and were opaque on the flat surfaces. The edges were polished clear using CeO_2 of a finer grit. The samples were first etched at room temperature for 24 hrs in a proprietary isotropic-HF etchant to remove 20 μm. This step assured that most of the microcracks and flaws occurring at edge were removed. A batch of six samples was mounted to a substrate holder, weighing 750 gm, using wax. Each face of the samples was first polished using a 2 % (by weight) TiO_2-deionized H_2O slurry on a tin lap until 35 μm was removed. The TiO_2 particle size is approximately 1000 Å and tends to conglomerate in the slurry, resulting in an abrasive action. The removal rate in this process was roughly 1 μm/hr. The samples were then float polished in a 10 % (by volume) deionized H_2O-colloidal SiO_2 suspension (with 10 Å particles) on a nylon lap. With particles as small as 10 Å float polishing is easily accomplished.

The samples were then tested for stress failure threshold in the apparatus shown in Figure 2, in which a three point loading is obtained, forcing maximum stress along the center line of a sample. The crystals were oriented on the apparatus so that the easy-cleavage plane was perpendicular to the direction of maximum stress imposed by the testing fixture. The stress failure results are shown in Figure 3. 40 % of the samples tested, fractured at stresses of 60-70 Klb/in^2, and approximately 10 % had fracture thresholds of 80-90 Klb/in^2. By observing the broken parts in an SEM, it was proposed that a majority of the resonators fractured from stress arising at the edge due to the testing procedure. There were no apparent fractures arising from microcracks in the surface or the edge. This is an indicator that most of the sub-surface damage layer

was removed in the fabrication process, and the stress fracture threshold was increased 4-10 times (approaching the bulk value) that of conventionally polished samples.

The future direction of this polishing research is an analysis of the sub-surface damage layer in float polished quartz and fused silica substrates. The substrates will be prepared using a similar procedure as above, and will be analyzed using optical scatter and photo-acoustic spectroscopy. In addition Nomarski and total internal reflection microscopy will be used to characterize the surfaces. Polishing techniques for materials such as ferrite and zinc sulfide will also be developed.

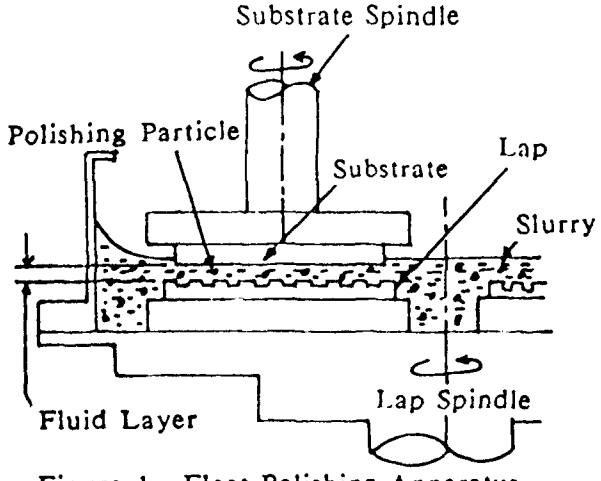

Figure 1. Float Polishing Apparatus.

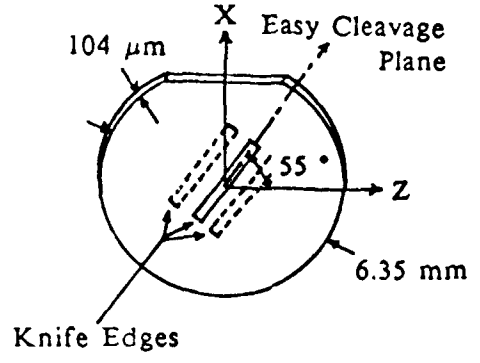

Figure 2. Quartz Resonator and Stress Threshold Test Apparatus.

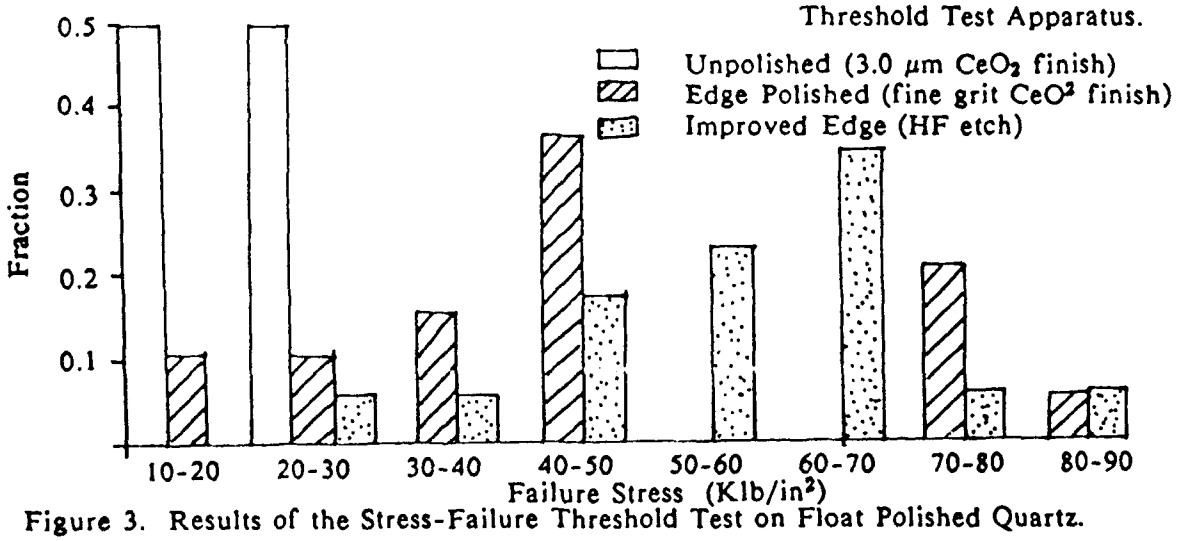

Figure 3. Results of the Stress-Failure Threshold Test on Float Polished Quartz.